

TRANSLATION

- (19) Patent Bureau of Japan (JP)  
 (12) Official Gazette for Unexamined Patents (A)  
 (11) Kokai Patent Number: Sho 63 (1988)-232814  
 (48) Disclosure Date: September 28, 1988

<u>Int. Cl<sup>4</sup></u>	<u>Identification Symbol</u>	<u>Intrabureau No.</u>
B 01 D 39/18		6703-4D
39/14		A-6703-4D
39/16		A-6703-4D
D21 H 5/18		D-7003-4L
		G-7003-4L

Request for Examination: Not Requested  
 Number of Invention: 1 (Total of 3 Pages)

(54) PRECISION FILTER PAPER

- (21) Application No.: Sho 62 (1987)-65990  
 (22) Application date: March 20, 1987  
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SPECIFICATION

1. Title of Invention  
 Precision Filter Paper
2. Claims  
 1 Precision filter paper for liquid treatment prepared by sheeting a fiber mixture comprizing 5 ~ 50 wt % of organic synthetic

fibers having the fiber diameter of below  $0.7\ \mu\text{m}$  and 50 ~ 95 wt % of fibers having the fiber diameter of more than  $2\ \mu\text{m}$ .

2 The precision filter paper for liquid treatment as described in Claim 1 in which the organic synthetic fiber having the fiber diameter of below  $0.7\ \mu\text{m}$  can be obtained from a stiff (illegible) synthetic polymer.

3 The precision filter paper for liquid treatment as described in Claim 2 in which the organic fiber having the fiber diameter of below  $0.7\ \mu\text{m}$  obtainable from a stiff (illegible) synthetic polymer is a micro-fibrillated fiber.

### 3. Detailed Description of the Invention

#### [Field of Industrial Applications]

The present invention relates to precision filter paper for liquid treatment, and relates to the filter paper outstanding in capturing particulates in liquid.

#### [Prior Art and Problems Thereof]

When improvement in the performance of filter paper for liquid treatment to capture particulates is desired, the objective can be attained by increasing the degree of beating of cellulose pulp. However, since (illegible) deteriorates at the same time, paper-making takes a long time, productivity falls, filtration resistance of the filter paper also increases, and filtration also takes a long time. In filter paper, thinning the constitutive fiber can improve the performance of capturing. However, when hydrophilic fibers like wood pulp are subject to beating, their thinning takes place simultaneously with flattening, they swell in water during paper-making, fibers in contact with each other remain adhered even after drying, and they tend to increase filtration resistance.

#### [Means to Solve the Problems]

It is mentioned above that, when cellulose pulp is subject to beating by the usual method, thinning takes place simultaneously with flattening. On the other hand, it was discovered that when a pulp slurry dispersed in water was allowed to pass at a high speed through an orifice before and behind which a pressure difference more than  $200\ \text{Kg}/\text{cm}^2$  was arranged, and a shear force was applied to slurry particles by reducing the speed at once, the pulp became microfibrils in which the pulp was finely divided in the fiber axis direction (Japanese Kokai Patent Sho 56 (1981)-100801). When this process is repeated and applied to a pulp slurry, cellulose pulp is obtained as microfibrils having the diameter of below  $0.7\ \mu\text{m}$ . When this micro-fibrillated cellulose is used as the constitutive component of filter paper, the ability to capture improves. However, in the case of cellulose, swelling in water is great, and although there is no flattening filtration resistance still tends to increase.

The present inventors applied the above-mentioned method of micro-fibrillation to a certain type of synthetic polymer fiber. As a result, the present inventors discovered that, since micro-fibrillation became possible and neither flattening nor swelling in water took place,

the paper that used this micro-fibrillated synthetic polymer as the constitutive component could improve the performance of capturing without increasing pressure loss. Thus, the present inventors achieved the present invention.

[Constitution of the Invention]

That is to say, the present invention relates to precision filter paper for liquid treatment prepared by sheeting a fiber mixture comprising 5 ~ 50 wt % of organic synthetic fibers having the fiber diameter of below 0.7  $\mu\text{m}$  and 50 ~ 95 wt % of fibers having the fiber diameter of more than 2  $\mu\text{m}$ .

For the organic synthetic fiber having the fiber diameter of below 0.7  $\mu\text{m}$ , preferably it is as stiff as possible; in particular, the fiber comprising a material known generically as stiff (illegible) synthetic polymer is suitable. Stiff (illegible) synthetic polymer means a polymer which maintains linear chains in solution having the chain length of more than 50 Å. For example, they are poly (p-phenyleneterephthalamide), poly (p-benzamide), poly (p-phenylenebenzobisthiazole), poly (p-phenylenebisoxazole), poly (amidohydrazide), polyhydrazide and poly (p-phenyleneterephthalamide-3,4-diphenyl ether terephthalamide). The stiff (illegible) synthetic polymer is once spun to fiber, or its chips are beaten to the pulp state, then it is micro-fibrillated with the above-mentioned method in which it is allowed to pass at a high speed through an orifice having a pressure difference, and it can be obtained as a bundle of micro-fibrillated fibers having the diameter of below 0.7  $\mu\text{m}$ . Since these fibers have good orientation of molecular chains, dividing in the fiber axis direction is also easy.

The micro-fibrillated fibers of these stiff (illegible) synthetic polymers do not swell in water, and the effect of flattening is also very small. Hence, when they are used for the constitutive component of paper, the performance to capture particles can be improved without increasing resistance to filtration. Furthermore, when paper-making is undertaken from an aqueous slurry, the micro-fibrillated fiber of stiff (illegible) synthetic polymer can maintain its shape by a proper (illegible), and articles that can be used as filter paper without special addition of a binder can be obtained.

When hydrophilicity of organic fibers is not great, as long as fibers having the diameter of below 0.7  $\mu\text{m}$  can be obtained by other methods, they can be used as the constitutive component of the precision filter paper of the present invention. Namely, fibers having a fine size can be obtained also from super-fine fibers out of split conjugated fibers obtained from the conjugated spinning method, whiskers obtained by allowing a monomer to polymerize and crystallize, fine splits from film, and the flash spinning obtained by allowing a spinning solution to spurt under a shear force. These fibers can also be used for the filter paper of

the present invention as long as their diameter is below  $0.7\ \mu\text{m}$ . However, generally because of the lack of maintenance of the sheet form like micro-fibrillated fibers, in many cases a binder is required for paper-making.

Suitable for a binder to maintain the form of filter paper and to increase the strength are an aqueous solution of thermosetting polyamide, acrylic resin and epoxy resin; when added to a fiber slurry during paper-making, preferable are those which undergo cross-linking when a sheet is heated and dried by heat-rolling.

Suitable for the fiber having the diameter more than  $2\ \mu\text{m}$  may be natural fibers ranging from cellulose fibers; synthetic polymer fibers may also be suitable. Furthermore, inorganic fibers such as asbestos, alumina, beryllium oxide, boron carbide, silicon carbide, nitrogen carbide, potassium titanate, graphite and silica may also be suitable. Furthermore, adoptable is also a method in which part of the fiber having the diameter of more than  $2\ \mu\text{m}$  is allowed to be a heat-weldable fiber and the fibers are allowed to be bonded during heat-roll processing.

In the precision filter paper for liquid treatment of the present invention, the performance of capturing particles becomes higher with increasing amount of fibers having the fiber diameter of below  $0.7\ \mu\text{m}$ , but raw material cost rises and paper strength also falls. Therefore, 5 ~ 50 wt % of the total amount of fiber is a preferable range.

[Effect of the Invention]

The precision filter paper of the present invention is a filter paper for liquid treatment excellent in the ability to capture particulates. In particular, the filter paper obtained from paper-making without using a binder, in which micro-fibrillated fibers of stiff (illegible) polymer are used as the fiber having the diameter of below  $0.7\ \mu\text{m}$ , and inorganic fibers or an organic stiff (illegible) polymer is used for the fiber having the diameter of more than  $2\ \mu\text{m}$ , exhibits high chemical resistance and also heat resistance. Hence, it can be utilized for various applications.

[Examples of Embodiment]

Examples of embodiment are used to describe the present invention concretely in the following. However, the present invention is not limited to them.

Example 1

Kevlar fiber (pulp grade product made by DuPont Co.; cut to 3 ~ 4 deniers and fiber length of ca. 2 mm) 10 g was dispersed in water 1 l, charged into the homogenizer (made by Gaulin Co. 15 M-STA) at ca.  $25\ ^\circ\text{C}$ , and allowed to (illegible) pass 30 times through an orifice with the pressure difference of  $420\ \text{Kg}/\text{cm}^2$  to obtain a uniform suspension of micro-fibrillated fibers of 1 % solids content. The diameter of micro-fibrillated fibers in the suspension was ca.  $0.4\ \mu\text{m}$ .

A mixed slurry of the above-mentioned homogenized suspension 80 wt parts, a 1 % aqueous suspension of Kevlar short fiber [3 ~ 4 d (ca. 20 ~ 24 $\mu$ m), fiber length ca. 3 mm] 2000 wt parts and water 2000 wt parts was introduced to the standard angle manual paper-making machine (wire 80 meshes) and (illegible) to obtain a filter paper of 75 g/m<sup>2</sup> basis weight. Physical values and filtration performance were measured and the results are shown in Table 1.

#### Comparative Example 1

The same measurements as those of the filter paper obtained in Example 1 were made on a commercially available filter paper for precision filtration.

#### Comparative Example 2

(Illegible) wood pulp 10 g was dispersed in water 1 l to prepare a 1 wt % slurry, charged into the homogenizer (made by Gaulin Co., 15 M-STA) at ca. 25°C and allowed to (illegible) pass 30 times through an orifice with a pressure difference of 420 Kg/cm<sup>2</sup> to obtain a micro-fibrillated cellulose suspension. The diameter of micro-fibrillated cellulose in the suspension was ca. 0.4  $\mu$ m.

A slurry comprizing a micro-fibrillated cellulose suspension 80 wt parts, a 1 % aqueous suspension of Kevlar short fiber 2000 wt parts and water 2000 wt parts was subject to paper-making with the same method as that in Example 1 to obtain a filter paper.

#### Example 2

Used in place of Kevlar short fiber in Example 1 was a glass short fiber (Asahi Fiber Glass, Mild Fiber-MF-B 10  $\mu$ m diameter, 200  $\mu$ m fiber length). Otherwise, the rest was carried out in the same way to obtain a filter paper.

Table 1

	Ex. 1	Ex. 2	Comp. Ex. 1	Comp. Ex. 2	Comp. Ex. 3
Basis wt (g/m <sup>2</sup> )	75	75	300	75	75
Filtration time (sec.)	14	10	160	2400	1500
0.5 $\mu$ m particles interception (%)	100	100	20	80	85

Filtration time: Time required to filter 100 ml of an aq. suspension of 0.5  $\mu$ m polystyrene standard

particles 10 ppm with a circular  
filter of 4 cm diameter.  
Interception: Degree of interception by the above-mentioned  
operation.

Comparative Example 3

Used in place of Kevlar short fiber of Comparative Example 2  
was a glass short fiber (Asahi fiber glass, Mild Fiber MF-B, 10  $\mu$ m  
diameter, 200  $\mu$ m fiber length). Otherwise, the rest was carried out in  
the same way to obtain a filter paper.

Translation: Language Services  
C & G Associates  
March 23, 1998